

WHAT IS CLAIMED IS:

1 1. A method of preparing ultra-high-purity
2 buffered-hydrofluoric acid or ammonium fluoride of
3 controlled concentration, comprising bubbling purified
4 ammonia vapor into ultra-pure hydrofluoric acid.

1 2. The method according to claim 1, wherein ultra-
2 high-purity buffered-hydrofluoric acid is prepared.

1 3. The method according to claim 2, wherein said
2 ultra-high-purity buffered-hydrofluoric acid has a
3 concentration of 10:1, 50:1 or 200:1 as measured by volume
4 parts of 40% ammonium fluoride to 49% HF.

1 4. The method according to claim 1, wherein ultra-
2 high-purity ammonium fluoride is prepared.

1 5. The method according to claim 4, wherein the
2 ammonium fluoride is a 40% by weight ammonium fluoride
3 solution.

1 6. The method according to claim 1, wherein the
2 ammonia vapor bubbling is performed in a generator which is
3 connected to a point of use.

1 7. The method according to claim 6, wherein the point
2 of use is located in a semiconductor device fabrication
3 facility.

1 8. The method according to claim 1, wherein the ultra-
2 pure hydrofluoric acid is prepared by a process comprising
3 the steps of:

4 removing a flow of hydrogen fluoride vapor from a
5 source of hydrogen fluoride;

6 contacting said hydrogen fluoride vapor with a
7 recirculating volume of high-purity water containing a high
8 concentration of hydrogen fluoride in a hydrogen fluoride
9 ionic purifier unit, wherein said hydrogen fluoride ionic
10 purifier unit passes purified hydrogen fluoride gas; and
11 combining the hydrogen fluoride gas with acidic
12 deionized water to produce the ultra-pure hydrofluoric acid.

1 9. The method according to claim 8, wherein the source
2 of hydrogen fluoride is an anhydrous hydrogen fluoride
3 source.

4 10. The method according to claim 8, wherein the
5 source of hydrogen fluoride is essentially arsenic-free.

6 11. The method according to claim 8, wherein the
7 source of hydrogen fluoride is ultra-pure arsenic-free
8 aqueous hydrogen fluoride.

9 12. The method according to claim 8, wherein the
10 ammonia vapor is prepared by a process comprising the steps
11 of:

12 removing a flow of ammonia vapor from a source of
13 liquid ammonia;

14 contacting said flow of ammonia vapor with a
15 recirculating volume of high-purity water containing a high
16 concentration of ammonium hydroxide in an ammonia ionic
17 purifier unit, wherein said ammonia ionic purifier unit
18 passes said purified ammonia vapor.

19 13. The method according to claim 12, wherein said
20 recirculating volume of high-purity water in said hydrogen
21 fluoride ionic purifier and said recirculating volume of
22 high-purity water in said ammonia ionic purifier are free of
23 additives.

1 14. The method according to claim 1, wherein the
2 ammonia vapor is prepared by a process comprising the steps
3 of:

4 removing a flow of ammonia vapor from a source of
5 liquid ammonia;
6 contacting said flow of ammonia vapor with a
7 recirculating volume of high-purity water containing a high
8 concentration of ammonium hydroxide in an ammonia ionic
9 purifier unit, wherein said ammonia ionic purifier unit
10 passes said purified ammonia vapor.

11 15. The method according to claim 1, wherein the step
12 of bubbling the purified ammonia vapor into the ultra-pure
13 hydrofluoric acid is performed in a generator, and wherein
14 the ultra-pure hydrofluoric acid is formed by introducing a
15 49% by weight hydrogen fluoride solution into the generator,
16 and diluting said hydrogen fluoride solution with
17 high-purity water.

18 16. The method according to claim 1, wherein
19 additional hydrofluoric acid is added to the solution after
20 the ammonia bubbling step, thereby forming said
21 ultra-high-purity buffered-hydrofluoric acid.

22 17. The method according to claim 16, wherein the
23 ammonia bubbling step forms a 40% ammonium fluoride solution
24 product.

25 18. The method according to claim 1, wherein the
26 ultra-pure hydrofluoric acid is formed by introducing
27 anhydrous hydrogen fluoride into high purity water in a
28 generator, and the ammonia vapor is bubbled into the ultra-
29 pure hydrofluoric acid in the generator.

30 19. The method according to claim 1, wherein the
31 concentration of the buffered-hydrofluoric acid or ammonium

fluoride is controlled by a step for detecting an endpoint of chemical mixing.

20. The method according to claim 19, wherein the step for detecting an endpoint of chemical mixing is performed by acoustic velocity measurement.

21. A system for preparing ultra-high-purity buffered-hydrofluoric acid or ammonium fluoride of controlled concentration, comprising a source of purified ammonia vapor, a source of ultrapure hydrofluoric acid and a generator which combines said ammonia vapor with said ultra-pure hydrofluoric acid to produce said ultra-high-purity buffered-hydrofluoric acid or ammonium fluoride.

22. The system according to claim 21, wherein the generator is connected to a point of use through piping.

23. The system according to claim 22, wherein the point of use is located in a semiconductor device fabrication facility.

24. The system according to claim 23, wherein the source of ultrapure hydrofluoric acid comprises a reservoir connected to receive a hydrogen fluoride source and to provide a flow of hydrogen fluoride vapor therefrom, said flow of hydrogen fluoride vapor being connected to pass through a hydrogen fluoride ionic purifier unit which provides a recirculating volume of high-purity water containing a high concentration of hydrogen fluoride in contact with said flow of hydrogen fluoride vapor, wherein said purifier passes purified hydrogen fluoride gas, and a hydrogen fluoride generator unit, connected to receive said flow of hydrogen fluoride gas from said purifier and to combine said hydrogen fluoride gas with high-purity acidic

14 deionized water to produce said ultra-pure hydrofluoric
15 acid.

1 25. The system according to claim 24, wherein the
2 source of hydrogen fluoride is an anhydrous hydrogen
3 fluoride source.

1 26. The system according to claim 24, wherein the
2 source of hydrogen fluoride is essentially arsenic-free.

1 27. The system according to claim 24, wherein the
2 source of hydrogen fluoride is ultra-pure arsenic-free
3 aqueous hydrogen fluoride.

1 28. The system according to claim 24, wherein the
2 source of purified ammonia vapor comprises a reservoir
3 connected to receive a liquid source of ammonia and to
4 provide a flow of ammonia vapor therefrom, said flow of
5 ammonia vapor being connected to pass through an ammonia
6 ionic purifier unit which provides a recirculating volume of
7 high-purity water, containing a high concentration of
8 ammonium hydroxide, in contact with said flow of ammonia
9 vapor, wherein said ammonia purifier passes said purified
10 ammonia vapor.

1 29. The system according to claim 24, wherein said
2 recirculating volume of high-purity water in said hydrogen
3 fluoride ionic purifier and said recirculating volume of
4 high-purity water in said ammonia ionic purifier are free of
5 additives.

1 30. The system according to claim 21, wherein the
2 source of purified ammonia vapor comprises a reservoir
3 connected to receive a liquid source of ammonia and to
4 provide a flow of ammonia vapor therefrom, said flow of
5 ammonia vapor being connected to pass through an ammonia

6 ionic purifier unit which provides a recirculating volume of
7 high-purity water, containing a high concentration of
8 ammonium hydroxide, in contact with said flow of ammonia
9 vapor, wherein said ammonia purifier passes said purified
10 ammonia vapor.

1 31. The system according to claim 21, further
2 comprising means for detecting an endpoint of chemical
3 mixing.

1 32. The system according to claim 31, wherein the
2 means for detecting an endpoint of chemical mixing comprises
3 an acoustic velocity measurement sensor.